Research article

JGS
Journal of the Geological Society

Published online May 15, 2017

https://doi.org/10.1144/jgs2016-119 | Vol. 174 | 2017 | pp. 836–849

LA-ICP-MS U-Pb dating of Carboniferous ash layers in the Cantabrian Zone (N Spain): stratigraphic implications

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Abstract: Seven centimetre-thick volcanic ash-fall layers interbedded within the thick Carboniferous successions of the Cantabrian Zone in northern Spain were dated by U–Pb zircon laser ablation inductively coupled plasma mass spectrometry across an interval ranging from Visean to Kasimovian, thus covering most of the Carboniferous period. All these ash layers occur in fossiliferous successions, allowing us to insert the radiometric data within a well-constrained biostratigraphic framework. Considering the analytical uncertainty, the obtained ages match the ages inferred from the conodont biostratigraphy established in the Mississippian succession (which hosts the oldest two ash layers, Visean in age), and the fusuline and mega- and microflora data from the strata hosting the Moscovian and Kasimovian (Westphalian–Stephanian) tonsteins. The age of a Langsettian tonstein along with data provided by several papers stating that in the Cantabrian Zone Langsettian floras were contemporaneous with lowermost Moscovian fusulines suggest that Langsettian floras could have been younger in Spain than in other areas. Our absolute ages provide new constraints not only for the correlation of the Carboniferous successions of the Cantabrian Zone with time-equivalent reference successions in other parts of the world but also for calibrating the Carboniferous global chronostratigraphic units based on marine fossils with the West European regional units.

Supplementary material: Stratigraphic position and biostratigraphic information on the studied ash-fall layers, analytical methods and geochronological results are available at https://doi.org/10.6084/m9.figshare.c.3768701

Received 11 October 2016; **revised** 26 January 2017; **accepted** 5 March 2017

The chronostratigraphic timescale is based on an attempt at precise correlation of worldwide stratigraphic successions, in which relevant biozones and boundaries of the chronostratigraphic units (relative scale) are well documented. In the Geological Time Scale published in 2012 (GTS2012; Gradstein *et al.* 2012) three Carboniferous and Permian composite stratigraphic standards were developed: (1) Donets Basin and Urals; (2) Guadalupian Mountains; (3) Lopingian composite section. The two last stratigraphic standards incorporate biostratigraphic information from several areas of the world (Davydov *et al.* 2012; Fig. 1a). The absolute ages assigned to stage, substage and biozone boundaries (mainly based on ammonoids, conodonts, fusulines and other benthic foraminifers) were estimated on the basis of available precise radiometric isotope dilution thermal ionization mass spectrometry (ID-TIMS) U–Pb zircon data published by Mundil *et al.* (2004), Ramezani *et al.* (2007), Gastaldo *et al.* (2009), Davydov *et al.* (2010) and Schmitz & Davydov (2012), which provided a re-scaling of the previous comparative standards (Davydov *et al.* 2012).

High-precision radiometric ages recently obtained from other Carboniferous successions (Pointon *et al.* 2012, 2014; Knight & Wagner 2014; Michel *et al.* 2015; Opluštil *et al.* 2016*a*,*b*) may give a higher precision to the Global Time Scale and improve the stratigraphic correlations. This is the aim of the study reported in this paper, in which the absolute ages of two Mississippian ash layers and five Pennsylvanian tonsteins intercalated within fossiliferous successions of the Cantabrian Zone (NW Spain) are given (see Fig. 1a and b). These radiometric ages, obtained by laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS), are intended to serve as a test for future high-precision ID-TIMS U–Pb dating. Coupled with the two ages of Stephanian B tonsteins by Knight & Wagner (2014), they provide new geochronological constraints for correlating the stratigraphic successions of the Cantabrian Zone with other Pennsylvanian areas of the world.

Interest of the Cantabrian Zone for Carboniferous correlation

The Carboniferous period was a time of rapid changes in global palaeogeography. The assemblage of Pangaea and the closure of the Rheic marine gateway, connecting the western Palaeothetys and the Panthalassa oceans, are interpreted to have caused a marked provincialism within the Pennsylvanian marine biota, which is the origin of long-lasting difficulties in establishing global correlations in the Carboniferous system. In addition to the correlation of the marine successions remaining difficult, the correlation of the chronostratigraphic scales between Western and Eastern Europe, largely based on terrestrial fossils, is equally contentious (e.g. Wagner & Winkler Prins 1994, 1997, 2016). In relation to both issues, the Carboniferous of the Cantabrian Zone shows characteristics that may help to resolve some of these problems.

(1) The Cantabrian Zone successions show a complete and continuous record of conodont-bearing rocks of Mississippian age (mostly Visean and Serpukhovian) and

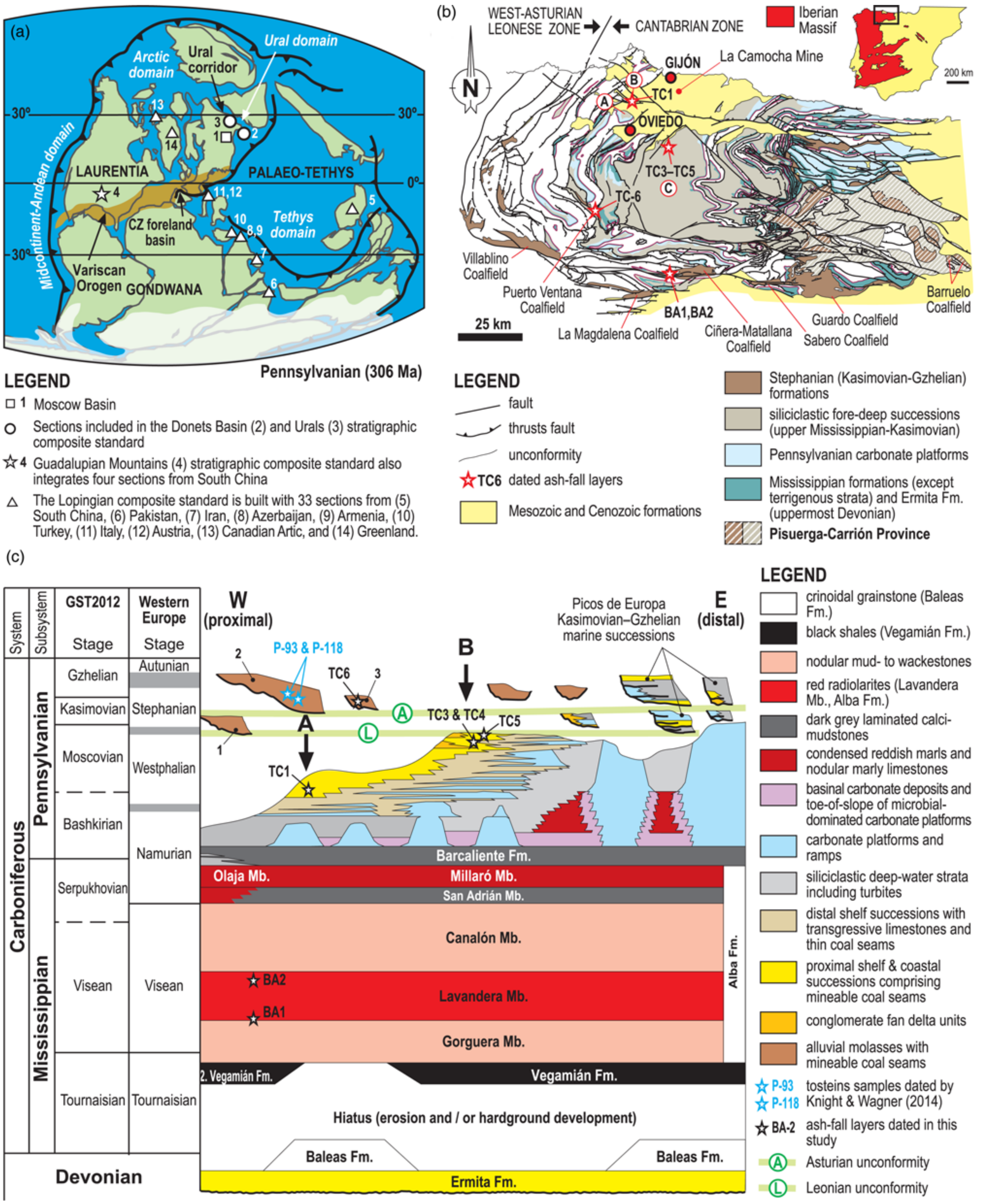

Fig. 1. (**a**) Palaeogeography of Pangaea during Pennsylvanian time with the location of the Variscan orogen and the foreland basin of the Cantabrian Zone (CZ) (modified after Scotese 2001; Golonka 2002). The location of the stratigraphic successions used to build the Donets Basin and Urals, Guadalupian and Lopingian composite stratigraphic standards and the four fusuline palaeobiogeographical domains defined by Rui *et al.* (1991) are indicated. (**b**) Schematic geological map of the Cantabrian Zone showing the distribution of Carboniferous successions with the location of the studied ash-fall samples (modified from Alonso *et al*., 2009). A, Naranco syncline; B, Santo Firme; C, Central Asturian Coalfield. (**c**) Synthetic chronostratigraphic diagram showing the stratigraphy of Carboniferous successions in the Cantabrian Zone (excluding the Pisuerga–Carrión Province) with indication of the ash-fall layers. Black arrows indicate the location of the stratigraphic sections shown in Figure 2 (A, Santo Firme, Naranco syncline and La Camocha Mine; B, Central Asturian Coalfield). 1, Guardo Coalfield; 2, Sabero, Ciñera–Matallana, La Magdalena and Villablino coalfields; 3, Puerto Ventana Coalfield. The position of the Leonian and Asturian unconformities is also indicated.

earliest Pennsylvanian age (Bashkirian) (e.g. Higgins 1974; Higgins & Wagner-Gentis 1982; García-López & Sanz-López 2002*a*,*b*; Sanz-López *et al.* 2004, 2006, 2007; Sanz-López & Blanco-Ferrera 2012, 2013). A conodont zonation is at present well established for the Mississippian and lower Pennsylvanian strata (Sanz-López & Blanco-Ferrera 2012, 2013), with a potential candidate to be selected as Global Stratotype Section and Point (GSSP) for the base of the Serpukhovian (Cózar *et al.* 2016), and a key stratigraphic section without sedimentary gaps that embraces the Mississippian–Pennsylvanian boundary and records the early evolution of *Declinognathodus noduliferus* (Sanz-López & Blanco-Ferrera 2013). In addition, conodonts have also been recorded from marine strata ranging in age from Bashkirian to Gzhelian (Méndez & Menéndez-Álvarez 1985; Méndez 2002, 2006).

(2) Fusuline-bearing strata are abundant in the Pennsylvanian of the Cantabrian Zone. Fusuline assemblages are typical of the Palaeotethys province and show strong affinities with North Africa (van Ginkel 1986) and Central Asia (Villa *et al.* 2002). However, these assemblages also share common elements with the Ural province (comprising areas significant in Carboniferous stratigraphy such as the Moscow Basin, the Urals and the Donets Basin). Their study has provided a fusuline biozonation, correlatable with equivalent intervals defined in the Moscow Basin, Urals and Donets Basin (van Ginkel 1965, 1971, 1973, 1987; Villa 1995; van Ginkel & Villa 1996; Merino-Tomé *et al.* 2006; Villa *et al.* 2015; Villa & Merino-Tomé 2016). Of particular interest is the Bashkirian–Moscovian transition, which in the Cantabrian Zone seems to contain a fossiliferous interval not recorded in the Moscow Basin (Villa & Merino-Tomé 2016), and the Moscovian–Kasimovian transition bearing a continuous record of *Protriticites* species (Villa *et al.* 2015).

(3) High subsidence rates in a succession of sedimentary basins have generated a fairly complete Pennsylvanian sedimentary record, overlapping across local tectonic breaks, to cover the greater part of the Westphalian and Stephanian time interval with a good fossil record of mega- and microflora. These characteristics have allowed the definition, in the Cantabrian Zone, of the following substages of the Western European chronostratigraphic scale: Asturian (formerly Westphalian D), Cantabrian, Barruelian (formerly Stephanian A) and Saberian (Stephanian A–B transition) (Wagner & Winkler Prins 1985*a*,*b*; Wagner *et al.* 2002; Wagner & Álvarez-Vázquez 2010; Knight & Wagner 2014). Megafloral biozones have been established that embrace the Westphalian to Autunian successions (Wagner 1984; Wagner & Álvarez-Vázquez 2010). In addition, the common intercalations of fusuline-bearing strata have allowed correlation of Western and Eastern European chronostratigraphic units (Sánchez de Posada *et al.* 2002*a*,*b*; Wagner & Álvarez-Vázquez 2010).

Geological setting; the Carboniferous successions of the Cantabrian Zone

The Cantabrian Zone contains the most complete Carboniferous successions of Iberia (Fig. 1b and c). During this period, the Cantabrian Zone was a marine foreland basin several hundreds of kilometres wide (Julivert 1978; Marcos & Pulgar 1982; Águeda *et al.* 1991; Bahamonde *et al.* 2015) that developed contemporaneously with the assemblage of the Pangaea supercontinent.

The Carboniferous succession commences with Mississippian condensed marine sediments, reaching a total thickness of up to 55 – 60 m, accumulated in the distal areas of the foreland basin (Sanz-López *et al.* 2006). From base to top they comprise crinoidal grainstones (Baleas Fm), black shales (Vegamian Fm), red nodular limestones, radiolarites and black laminated calcic-mudstones and marls (Alba Fm) (Fig. 1c). The Mississippian–Bashkirian boundary lies within the overlying Barcaliente Fm, which consists of black laminated limestones and reaches some 350 m in thickness (Figs 1 and 2).

Overlying the Barcaliente Fm, a succession of Bashkirian and Moscovian strata, sourced mainly from the erosion of the Variscan orogenic chain (Pastor-Galán *et al.* 2013), may reach 8000 m in thickness (Fig. 2). Very thick and mainly siliciclastic marine and coastal strata accumulated in the proximal subsiding sectors of the basin, generally arranged in cyclothems of tens to hundreds of metres thickness that commonly include marine limestones (deposited during marine transgressions) and delta-plain to alluvial deposits with coal seams (Águeda *et al.* 1991; Bahamonde *et al.* 2015). Pioneer studies of the stratigraphy of the Carboniferous of the Cantabrian Zone (Barrois 1882) subdivided the paralic successions into two informal stratigraphic units having a coal mining significance, the Lena and Sama Groups. These groups were later subdivided within each coalfield into a number of informal stratigraphic units called ‘paquetes mineros’ (=mining stratal packages), usually hundreds of metres thick and containing sets of partially correlated mineable coal seams (e.g. García Loygorri *et al.* 1971).

In the course of the Variscan deformation, unconformable mid-Moscovian to Gzhelian (Duckmantian–Stephanian B) sediments were accumulated overlying the Palentine (only recognized in the Pisuerga–Carrión Province), Leonian and Asturian unconformities (De Sitter 1959; Wagner 1959; Fig. 1c) within piggy-back basins developed atop advancing thrust nappes and ahead of the orogenic front (Alonso 1987; Merino-Tomé *et al.* 2006, 2007, 2009; Martín-Merino *et al.* 2014; Alonso *et al.* 2015). Alluvial and coastal sediments with local marine incursions were deposited at different times along the western and southern Cantabrian Zone while marine sedimentation continued in the eastern part of the Cantabrian Zone (Colmenero *et al.* 2002; Fernández *et al.* 2004; Wagner & Álvarez-Vázquez 2010). These strata contain the most complete lower Stephanian record in Europe (Wagner & Winkler Prins 2016).

Carboniferous ash layers in the Cantabrian Zone

Within the wide terminology used in the literature on ash layers or ash-fall layers, in this paper we use the terms K-bentonite in the sense of Fischer & Schmincke (1984) and tonstein as defined by Bohor & Triplehorn (1993). A number of volcanic ash-fall layers, mostly tonstein occurrences, have been described within the Carboniferous successions of the Cantabrian Zone (Figs 1c and 2). Regarding the Mississippian strata, Loeschke (1983) studied two ash-fall layers from the Alba Fm in the vicinity of Sabero village and some others were recognized later from the same stratigraphic unit and at the top of the undelying Vegamián Fm (García-López & Sanz-López 2002*a*).

Pennsylvanian tonsteins embedded within mined coal seams were discovered in both the Westphalian paralic successions (Prado 1964; García Loygorri *et al.* 1971; Galán Arias *et al.* 1984; Rodríguez Mateos 1994) and the Stephanian successions of the Puerto Ventana (Enadimsa 1981), Ciñera-Matallana (Bieg & Burger 1992) and Sabero coalfields (Knight 1983; Knight *et al.* 2000) (see Fig. 1b for location).

Ash-fall samples analysed in this study

In the course of the present investigations two ash-fall samples from Mississippian strata (samples BA-1 and BA-2) and five samples from Pennsylvanian tonsteins (samples TC-1 to TC-6) have been

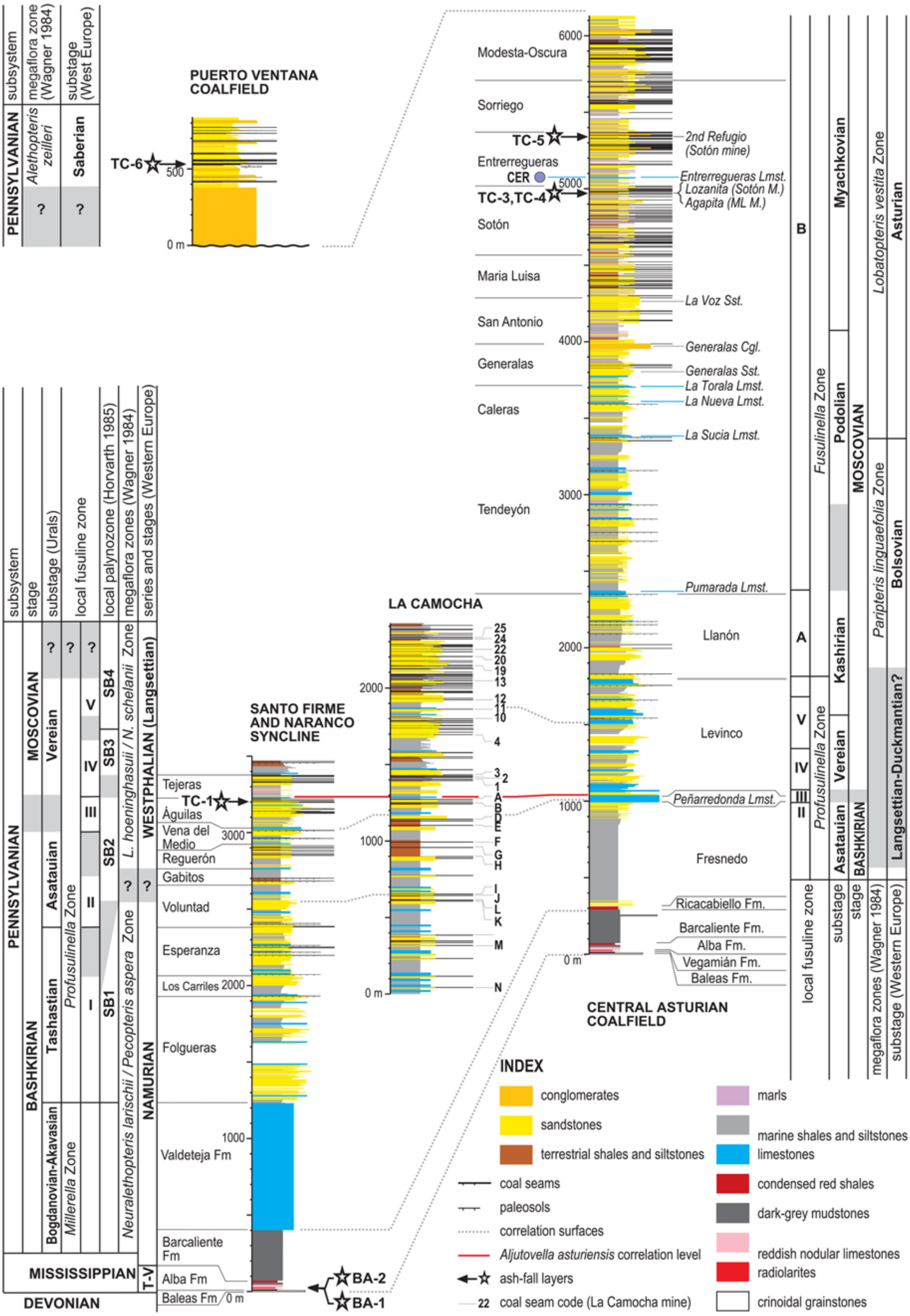

Fig. 2. Correlation of synthetic stratigraphic sections of the Santo Firme–Naranco syncline, La Camocha Mine and the Central Asturian Coalfield according to biostratigraphic information on fusulines (Villa & Merino-Tomé 2016) and the Puerto Ventana succession at the Bienvenida Mine. Santo Firme–Naranco syncline composite section is based on data of IGME (1984, 1985), ITGE (1987) and Trell *et al.* (1988). La Camocha Mine section is reconstructed using information from transects across 4th, 6th and 7th levels (IGME 1984) and Horvarth (1985); palynological zones are from Horvarth (1985) and stages and substages of Western European regional scale are according Wagner & Álvarez-Vázquez (1995). The Central Asturian Coalfield composite section is constructed using data from Salvador (1989, 1993), Barba (1991) and available information from Hunosa Mining Company. The Puerto Ventana section at the Bienvenida Mine is reconstructed using information from the 0, 1st and 2nd levels in the mine according Enadimsa (1981). Fusuline zones are based on Van Ginkel (1965, 1973, 1987), Villa (1995) and Villa & Merino-Tomé (2016), and megafloral zones are after Wagner & Álvarez-Vázquez (1991, 2010).

Table 1. *Summary of the characteristics of the volcanic ash-fall samples and ages with information on their geographical location, age and conodont, fusuline and megaflora zones*

Sample name	Sample type	Thickness (cm)	Location	Global stage, regional substage (Urals and Russian Platform); conodont or fusulinid zone	Regional stage and substage (Western Europe); megaflora zone	Concordia age (Ma); MSWD (of concordance); probability (*P*, of concordance)	Number of zircons dated
TC-6	Kaolinitic tonstein	3	Bienvenida 2nd Coal Mine, María Olga seam, Puento Ventana Coalfield (43°3.907′N, 5°59.879′W)		Lower Stephanian B (Saberian); *Alethopteris zeilleri* (Wagner & Álvarez-Vázquez 2010)	304.2 ± 1.1; MSWD = 0.14; *P* = 0.71	21
TC-5	Kaolinitic tonstein	1	Pozo Sotón, 2nd Refugio coal seam (El Entrego, San Martin del Rey Aurelio), Central Asturian Coalfield (43° 16.855′N, 5°37.304′W)	Moscovian (mid–late Myachkovian); *Fusulina alvaradoi* (this work)	Asturian; *Linopteris obliqua* (Wagner & Álvarez-Vázquez 1991, 2010)	307.12 ± 0.94; MSWD = 0.0015; *P* = 0.97	36
TC-4	Kaolinitic tonstein	2	Pozo Sotón (El Entrego, San Martin del Rey Aurelio), Lozanita coal seam, Central Asturian Coalfield (43°16.855′N, 5°37.304′W)	Moscovian (mid-Myachkovian); *Fusulina alvaradoi* (this work)	Asturian; *Linopteris obliqua* (Wagner & Álvarez-Vázquez 1991, 2010)	307.07 ± 0.90; MSWD = 0.56; *P* = 0.45	23
TC-3	Kaolinitic tonstein	2	Pozo Maria Luisa (Ciaño, Langreo), Agapita coal seam; Central Asturian Coalfield (43°17.330′N, 5°39.601′W)	Moscovian (mid-Myachkovian); *Fusulina alvaradoi* (this work)	Asturian; *Linopteris obliqua* (Wagner & Álvarez-Vázquez 1991, 2010)	307.7 ± 1.3; MSWD = 0.19; *P* = 0.66	29
TC-1	Kaolinitic tonstein	6	Minona Coal Mine (Posada de Llanera, Asturias), Aguila 4th coal seam; Santo Firme Coalfield (43°27.261′N, 5° 50.517′W)	Transition interval Bashkirian–Moscovian; *Profusulinella* Zone III (Villa & Merino-Tomé 2016)	Langsettian; *Lyginopteris hoeninghausii–Neuralethopteris schlehanii*	314.4 ± 1.3; MSWD = 0.23; *P* = 0.63	28
BA-2	Bentonite	5 – 10	Baleas Quarry (Pola de Gordón, León) (42° 51.946′N, 5°40.061′W)	Early Visean; *Gnathodus praebilineatus*		337.0 ± 1.0; MSWD = 0.60; *P* = 0.44	44
BA-1	Bentonite	3 – 5	Baleas Quarry (42° 51.949′N, 5°40.074′W)	Early Visean; *Gnathodus texanus*		343.5 ± 1.1; MSWD = 0.60; *P* = 0.44	25

dated. A detailed description of the stratigraphic position of the studied samples and available biostratigraphic data are given in the supplementary material and Table 1.

Samples BA-1 and BA-2 were collected in the Las Baleas quarry (Pola de Gordón, north of León; Fig. 1b) from two ash layers located respectively at the base and near the top of the Lavandera Member of the Alba Fm (see Figs 1c and 2 and supplementary material).

Sample TC-1 was collected by Prado (1964) from a tonstein embedded within the Águila 4th coal seam of the Águilas package (Fig. 2), exploited at the abandoned Minona Mine in the Santo Firme Coalfield (Fig. 1b). Samples TC-3 and TC-4 come from a tonstein layer located at the top of the Sotón stratal package that has been used as a key bed to correlate along the Central Asturian Coalfield (García Loygorri *et al.* 1971; Figs 1b and 2). It must be noted, however, that the host coal seam has been given different local names. Sample TC-3 was collected within the Agapita coal seam in the Pozo Maria Luisa Mine and sample TC-4 comes from the Lozanita coal seam in the nearby Pozo Sotón Mine. Sample TC-5 was collected from a tonstein layer described by Rodríguez Mateos (1994) within the 2nd Refugio coal seam located close to the top of the Entrerregueras stratal package in the Pozo Sotón Mine (Fig. 2). Sample TC-6 was collected by J. Prado from the María Olga coal seam in the Bienvenida 2nd Mine, Puerto Ventana Coalfield (Figs 1b and 2).

U–Pb results

A detailed description of the U–Pb LA-ICP-MS analytical procedure is provided in the supplementary material.

K-bentonite BA-1 (Visean, early Livian)

Sixty zircons from sample BA-1 were analysed, of which half yielded a concordant age. Twenty-five of the concordant zircons provide a coherent group that was used to calculate a concordia age of 343.5 ± 1.1 Ma (Fig. 3 and Table 1), which is interpreted as the age of the ash-fall layer. The other five concordant zircons that yielded older ages are interpreted as antecrysts and/or xenocrysts or inherited zircons.

K-bentonite BA-2 (Visean, late Livian)

Sixty zircons from sample BA-2 were analysed, of which 51 yielded a concordant age. Among the latter, 44 provided a coherent group used to calculate a concordia age of 337 ± 1 Ma (Fig. 3 and Table 1), which is interpreted as the age of this ash-fall layer. The other seven concordant zircons yielded older ages ranging from 362 to 393 Ma.

Tonstein TC-1 'Santo Firme' (Moscovian, early Vereian)

Forty-seven zircons from sample TC-1 were analysed and for 13 of them two separate analyses were performed. Thirty analyses yielded a concordant age and five of the zircons analysed twice provided identical concordant ages. The 17 concordant analyses with discordance <3% were used to calculate a concordia age of 314.4 ± 1.3 Ma (Fig. 3 and Table 1), which is interpreted as the age of the Santo Firme ash-fall layer.

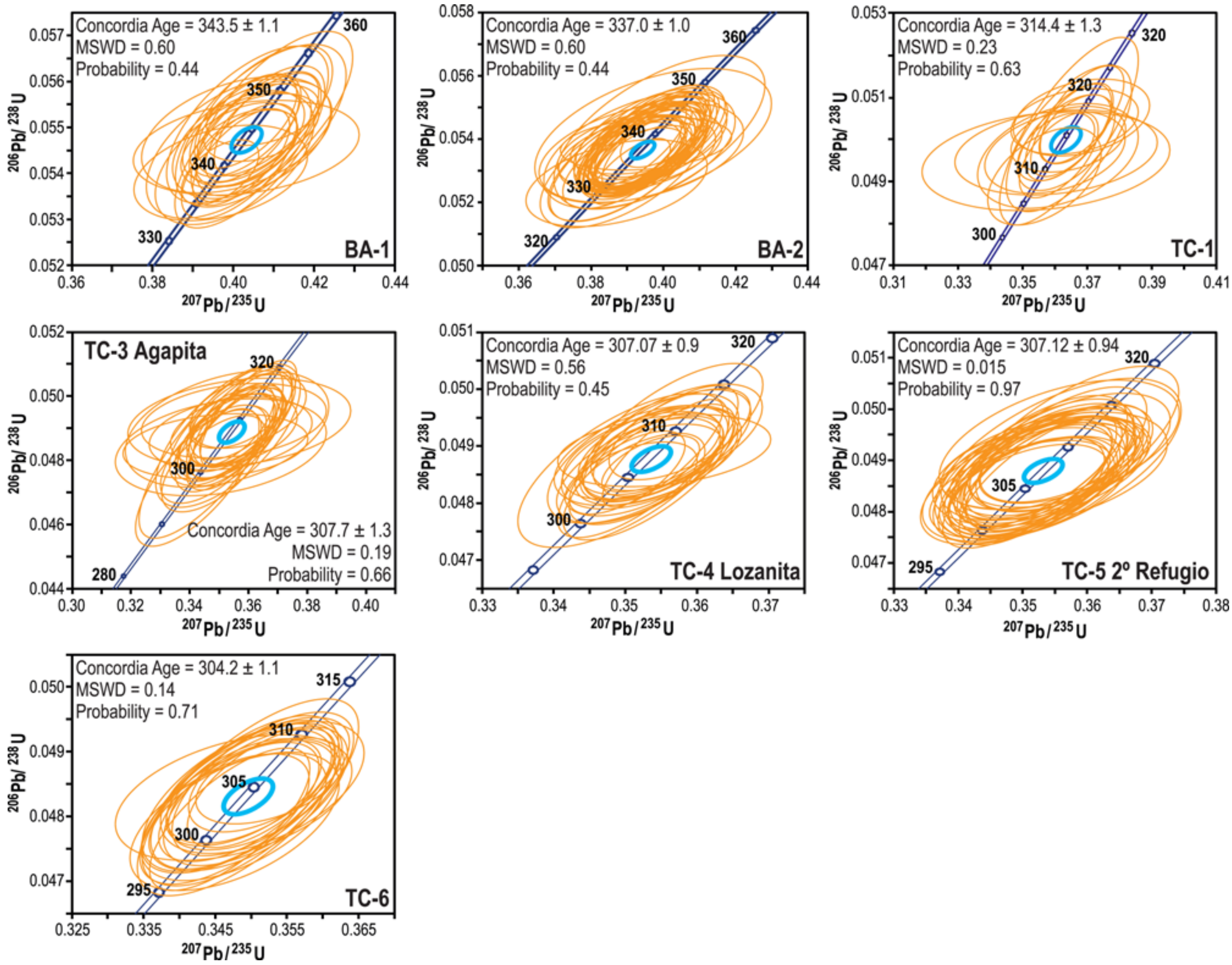

Fig. 3. Concordia plots for the studied ash-fall samples. Data-point error ellipses are 2σ, and 2σ decay-constant errors are included.

Tonsteins TC-3 'Agapita' and TC-4 'Lozanita' (Moscovian, early-mid Myachkovian, Asturian)

Sixty zircons were analysed and 33 yielded concordant ages in TC-3, and 23 in TC-4. The concordia age for TC-3 was obtained using a group of 29 coherent concordant analyses, with a result of 307.7 ± 1.3 Ma (Fig. 3 and Table 1). The 23 concordant ages in sample TC-4 provided a concordia age of 307.1 ± 1 Ma (Fig. 3 and Table 1).

Tonstein TC-5 '2nd Refugio' (Moscovian, late Myachkovian, Asturian)

From the 60 zircons analysed in this sample, 36 provided concordant ages and all were used to obtain a concordia age of 307.1 ± 1 Ma (Fig. 3 and Table 1). This age (late Myachkovian, but not latest) is consistent with the ages obtained for samples TC-3 and TC-4. However, the stratigraphic position of tonstein TC-5 is *c.* 350 m stratigraphycally higher than TC-3 and TC-4. The implications of this observation will be discussed in the following section.

Tonstein TC-6 'María Olga'(Stephanian B)

Sixty analyses were performed on 49 zircons, of which 21 yielded concordant ages and were used to obtain a concordia age of 304.2 ± 1.1 Ma (Fig. 3 and Table 1).

Discussion on the age and correlation of the Cantabrian Zone tonsteins

The U–Pb ages obtained in this study are robust and provide the first absolute geochronological dates for the Mississippian and early to mid-Pennsylvanian (Moscovian) successions of the Cantabrian Zone (Fig. 4).

Age of samples BA-1 and BA-2

The ages obtained are in agreement with the available biostratigraphic information. The age of sample BA-1 (343.5 ± 1.1 Ma) corresponds to the middle part of the *Gnathodus texanus* zone in GST2016 (Ogg *et al.* 2016), which is in accordance with the age assigned to the base of the Lavandera Member of the Alba Fm by García-López & Sanz-López (2002*a*) and Sanz-López & Blanco-Ferrera (2012). The age of sample BA-2 (337 ± 1 Ma) is in agreement with a position in the *Gnathodus praebilineatus* zone, as established in GST2012 (Davydov *et al.* 2012) and GST 2016 (Ogg *et al.* 2016).

Age of tonstein TC-1 and the Bashkirian–Moscovian boundary

Very high sedimentary rates approaching to 500 m Ma^{-1} (without taking into account the effects of sediment compaction) occurred in the Santo Firme area during the late Bashkirian. Tonstein layer TC-1 lies 190 m below the first reported occurrence of advanced *Verella*

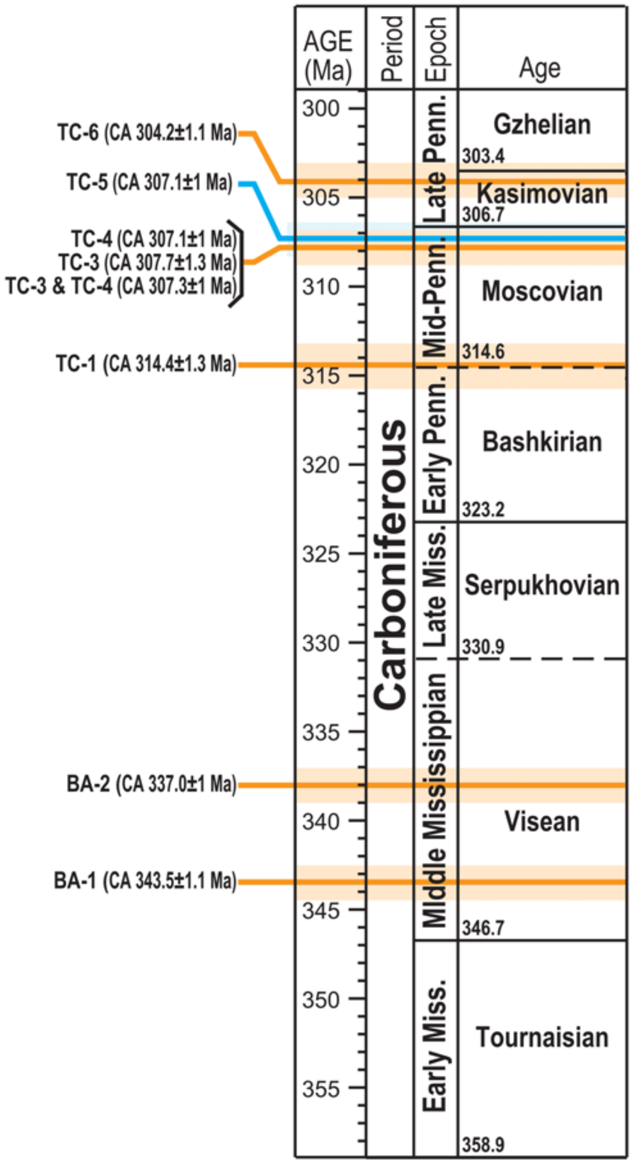

Fig. 4. Obtained ages of ash-fall layers in the Cantabrian Zone compared with the GST2016 (Ogg *et al.* 2016).

fusuline species (*V. transiens*; Villa & Merino-Tomé 2016), and thus it could be a maximum of 0.4 Ma older than this biostratigraphic event. In the absence of other fusuline species, *Verella transiens* has been considered in the Cantabrian Zone as a marker for the lowermost Moscovian (Villa 1995).

Considering the error ranges, the absolute age of the tonstein sample TC-1 (314.4 ± 1.5 Ma) is only slightly younger than the K_3 limestone in the Donets Basin (Davydov *et al.* 2010), where the first occurrence of the conodont *Diplognathodus ellesmerensis* (recently proposed to serve as a marker for the Bashkirian–Moscovian boundary; Ogg *et al.* 2016) is recorded, and the Bashkirian–Moscovian boundary (314.6 Ma, according to Davydov *et al.* 2010 and Ogg *et al.* 2016; 314.61 ± 0.33 Ma according to Peterson 2011). However, the age of TC-1 is around 1.6 Ma younger than the I_2^2 limestone (316 Ma according to Davydov 2009), where advanced *Verella*, and other fusuline species considered to be Moscovian in other Carboniferous areas, first occur in the Donets Basin (Tanaka & Ueno, cited by Alekseev & Task Group 2013) (Fig. 5). Therefore, if our date for sample TC-1 is correct, it would agree with the interpretation of Davydov (2009) that advanced *Verella* and other fusulines appeared in the Donets Basin earlier than in other areas of the world.

Age and correlation of tonsteins TC-3, TC-4 and TC-5

The Lozanita–Agapita tonstein level (samples TC-3 and TC-4) and the 2nd Refugio tonstein (sample TC-5) provided ages that fall within the Myachkovian, the same age as provided by brachiopods (Martínez-Chacón cited by Luque *et al.* 1985) and fusulines (van Ginkel 1973; this paper). The fusuline assemblage yielded by the Entrerregueras Limestone (sample CER, Fig. 2) consists of species belonging to the *Fusulina*, *Hemifusulina*, *Taitzehoella* and *Pseudostaffella (Neostaffella)* genera and suggests a correlation with a level within the N_1–N_1^2 interval in the Donets Basin (the *Hemifusulina graciosa–Fusiella spatiosa* Zone defined by Khodjanyazova & Davydov 2013) (Fig. 5). With respect to the Moscow Basin, the Entrerregueras Limestone could probably be correlated with the middle Myachkovian Domodedovo Fm.

Taking into account that the mean subsidence rates (Águeda *et al.* 1991) and sedimentation rates (without considering the effects of sediment compaction) estimated for the Asturian stage in the Central Asturian Coalfield were as high as than 950 m Ma^{-1} (see SP-Table 2), the Agapita–Lozanita tonstein (samples TC-3 and TC-4) might be ≈0.1 Ma older than the Entrerregueras Limestone, which is located 100 m above this ash-fall layer (Fig. 2). In the same way, the TC-5 (2nd Refugio tonstein), lying 250 m above the Entrerregueras Limestone, might be slightly less than 0.3 Ma younger than the aforementioned limestone bed. Our age for sample TC-3 matches the ages estimated for the limestones N_1, N_1^1 and N_1^2, which range from 307.6 to 307.8 Ma (Davydov *et al.* 2010), and the age of sample TC-4 is only 0.7 Ma younger. The age of sample TC-5 is slightly younger than the ages estimated for these limestones and very close to the age of the n_1 coal seam (307.26 ± 0.1 Ma) (Davydov *et al.* 2010). Thus our ages are compatible, considering the error ranges, with the ages estimated on the basis of available biostratigraphic data on fusulines (Fig. 5). It is also remarkable that the ages obtained for the Agapita–Lozanita and 2nd Refugio tonsteins are very close to the possible ages estimated for the base of the Kasimovian: 306.65 Ma (Davydov *et al.* 2010), 306.17 ± 0.49 Ma (Peterson 2011), 307.0 Ma (Davydov *et al.* 2012; Gradstein *et al.* 2012) or 306.7 Ma (Ogg *et al.* 2016) (see Fig. 4). Therefore, these ages might be relevant for future estimations of the absolute age of the Moscovian–Kasimovian boundary.

Concerning the West European timescale, both the Agapita–Lozanita tonstein (TC-3 and TC-4) and the 2nd Refugio tonstein (TC-5) fall within the upper part of the *Linopteris obliqua* megafloral zone of early Asturian age (Wagner 1984; Wagner & Álvarez-Vázquez 1991, 2010). The ages obtained in the present study are considered broadly compatible, taking account of error ranges, with the chemical abrasion (CA)-TIMS ages published for two tonsteins from central and western Bohemia in the Czech Republic of Asturian (Westphalian D) age (Opluštil *et al.* 2016*a*): 308.00 ± 0.04 Ma (sample 12) and 307.23 ± 011 Ma (sample 11). Both tonsteins fall within the *Lobatopteris vestita* Zone of Wagner (1984) and Wagner & Álvarez-Vázquez (2010) (the present authors note the systematic revision of *L. vestita* published by Wittry *et al.* (2015), and the usage by Opluštil *et al.* (2016*a*) of the designation *Crenulopteris acadica* Zone in place of the *L. vestita* Zone. As these modifications have implications with respect to the concept of the *Lobatopteris vestita* Zone (see discussion by Wagner & Álvarez-Vázquez 2016), it is considered appropriate here to apply the name and usage of this zone as defined by Wagner (1984) and Wagner & Álvarez-Vázquez (2010)). With respect to the age relationship, which may be assumed between the Bohemian tonsteins in the *L. vestita* Zone and the two Asturian Coalfield tonstein horizons (TC-3–TC-4 and TC-5) in the underlying *L. obliqua* Zone, it appears that the Asturian Coalfield samples give a younger age than would be expected, with a discrepancy of *c*. 1 My, and thus within the error ranges (Fig. 6).

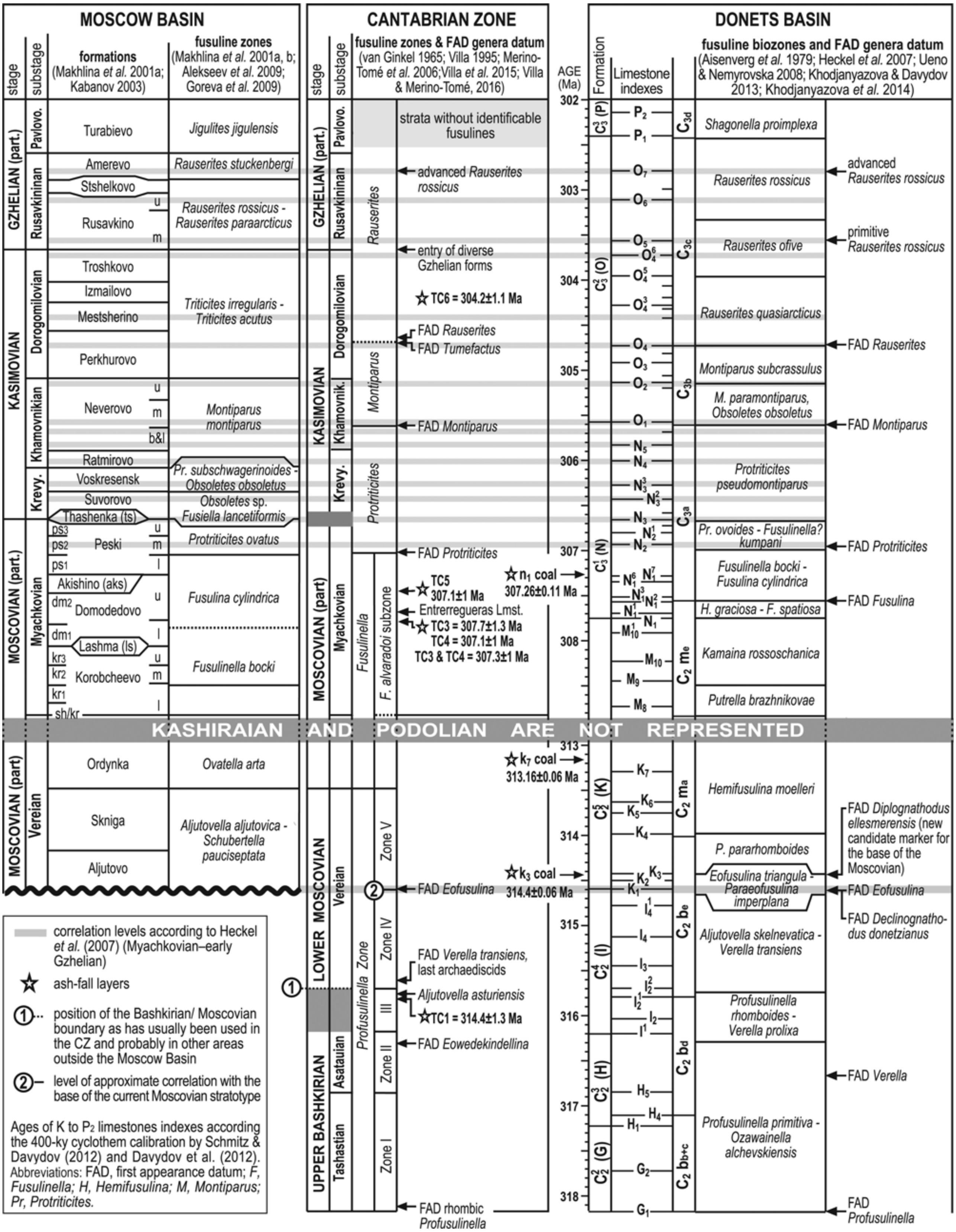

Fig. 5. Correlation of late Bashkirian–early Moscovian and Myachkovian–early Gzhelian fusuline biozones of the Cantabrian Zone, Moscow Basin and Donets Basin with the new U–Pb LA-ICP-MS dates of the Cantabrian Zone tonsteins presented in the present paper and ID-TIMS U–Pb ages of k_3, k_7 and n_1 tonsteins taken from Davydov *et al.* (2010). Equivalent biozones and fusuline biotic events are placed at the same horizontal level, according to Villa & Merino-Tomé (2016) for the late Bashkirian–early Moscovian, and following the digital cyclothem correlation proposed by Heckel *et al.* (2007) for the Myachkovian–early Gzhelian. Formations and fusuline ages in the Moscow Basin after Makhlina *et al.* (2001*a*,*b*), Kabanov (2003), Alekseev *et al.* (2009) and Goreva *et al.* (2009). Fusuline biozones and first appearance datum of fusuline genera in the Donets Basin after Aisenverg *et al.* (1979), Heckel *et al.* (2007), Ueno & Nemyrovska (2008), Khodjanyazova & Davydov (2013) and Khodjanyazova *et al.* (2014).

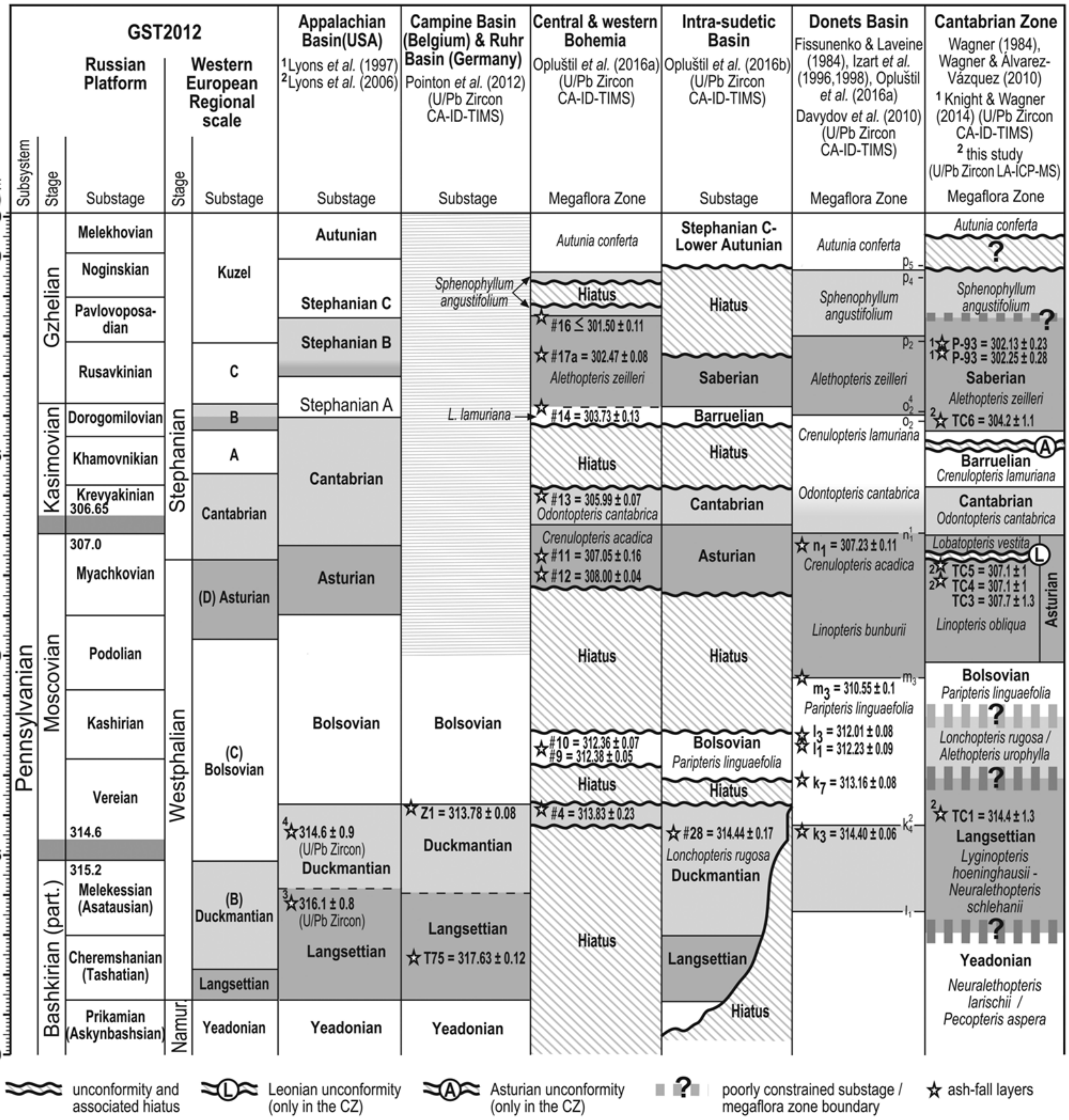

Fig. 6. Ages of North American and Western European regional stages and substages in the Appalachian Basin, Belgium, Germany, central and western Bohemia (Czech Republic), Intra-Sudetic Basin (Czech Republic), Donets Basin (Ukraine) and Cantabrian Zone (Spain) with published ages of tonsteins by Lyons *et al.* (1997, 2006), Davydov *et al.* (2010), Pointon *et al.* (2012), Knight & Wagner (2014) and Opluštil *et al.* (2016*a*,*b*), and the new U–Pb LA-ICP-MS dates of the Cantabrian Zone tonsteins presented in the present paper. The location (age) of tonsteins TC-3, TC-4 and TC-5 is represented considering the available biostratigraphic information on fusulines and megaflora. It is relevant to note the usage of Opluštil *et al.* (2016*a*) of the designation *Linopteris bunburii* Zone, following the terminology of Cleal (1991), but, as noted by Wagner & Álvarez-Vázquez (2010), this refers to the same index fossil recognized as *L. obliqua* var. *bunburii*, which is abundantly present in the Cantabrian Zone and characterizes the *L. obliqua* Zone.

Age of tonstein TC-6

A review of the Puerto Ventana flora by Wagner & Álvarez-Vázquez (2010) considered that the succession belongs to the *Alethopteris zeilleri* Zone, corresponding to the Saberian substage (Wagner & Álvarez-Vázquez 2010; Knight & Wagner 2014). The age of tonstein TC-6 (304.2 ± 1.1 Ma) is significantly older than the CA-TIMS ages of tonsteins 31 (sample P-118, 302.25 ± 0.28 Ma) and 32 (sample P-92, 302.13 ± 0.23 Ma) of Saberian age in the Sabero Coalfield (southern part of the Cantabrian Zone; see Fig. 1b) published by Knight & Wagner (2014). Nevertheless, considering the error range, our age is compatible with the age of 303.7 Ma estimated for the base of this megafloral zone in central and western Bohemia (Czech Republic) on the basis of the CA-ID-TIMS data published by Opluštil *et al.* (2016*a*) (sample 14: 303.73 ± 0.13 Ma). Our age is also close to the ages for coal No. 4 (304.07 ± 0.08 Ma) and coal No. 5 (303.95 ± 0.07 Ma) of the basal Graissessac Formation in the Lodève and Graissessac basins (southern France) determined by Michel *et al.* (2015) by means of CA-ID-TIMS. The floral assemblages characterizing this succession were interpreted as belonging to the Stephanian B by Becq-Giraudon (1973), and the flora recovered from the coal layers No. 3–7 has the same features as the upper Stephanian (Stephanian B + C) flora from the Saint-Étienne coal basin (Gand *et al.* 2013).

Accuracy of LA-ICP-MS dating

LA-ICP-MS zircon dating is a common and widespread method used to investigate the source areas of terrigenous deposits (Fernández-Suárez *et al.* 1999, 2014; Pastor-Galán *et al.* 2013; Shaw *et al.* 2014) and to constrain the ages of plutonic igneous rocks (Fernández-Suárez *et al.* 2011), and it is now commonly applied to tephrochronology to establish precise relative ages of volcanic ash layers (Bruguier *et al.* 2003; Bowring *et al.* 2006; Schaltegger *et al.* 2015). This technique allows the dating of single zircon crystals, separated from sample rocks, with a precision per analysis of 1 – 8% (2σ). This is lower than that for other analytical methods such as ID-TIMS or CA-TIMS (0.1 – 0.3% (2σ)) and similar to the precision per analysis achieved with secondary ion mass spectrometry (SIMS) (1 – 5% (2σ)) (Bowring *et al.* 2006). However, the advantages are that this method permits the analysis of a large population of zircon crystals in a single sample, allowing potentially different inherited zircon populations to be identified, and also allows statistically robust ages to be obtained using a significant number of concordant zircons. When a sufficient number of concordant zircons are used in a sample, generally more than 20, each with analytical errors of *c.* 1 – 5% (2σ), the concordia ages may provide ages with errors around 0.3%, as is the case in our results, in which all the composite errors are *c.* ±1 Ma, whereas single analysis errors range between *c.* ±4 and *c.* ±10 Ma.

In this study, the absolute ages obtained by means of LA-ICP-MS dating, considering the statistical error ranges, show a good match with the inferred ages estimated for each sample on the basis of the available biostratigraphic information. This suggests that a relatively high level of confidence can be placed on these ages. However, it is relevant to mention that the correlation of the stratigraphic successions of the Cantabrian Zone with the Donets Basin on the basis of fusulinid faunas suggests that the weighted mean ages (concordia ages) obtained for tonsteins TC-1, TC-4 and TC-5 are *c.* 0.3 – 1 Ma younger than might be expected, but still within the estimated error ranges.

Diachronism–isochronism of Westphalian and Stephanian floras

Micro- and megaflora have frequently been used as key fossil indices for biozonation of the Westphalian and Stephanian successions, particularly in those areas in which marine biota is absent or scarce. Recent high-precision dating of tonstein layers (e.g. Davydov *et al.* 2010; Pointon *et al.* 2012; Opluštil *et al.* 2016*a*,*b*) has provided valuable information for correlation of the West European chronostratigraphic units with the Russian Platform units. These radiometric data suggest that the base of the Duckmantian and Bolsovian substages could be relatively isochronous in the sedimentary basins located in the north side of the Variscan Orogen (Campine, Ruhr and intra-Sudetic basin and Appalachian basin) and the Donets Basin (Fig. 6). At variance with the above, Langsettian floras appear to be significantly younger in the Cantabrian Zone, where they are contemporaneous with latest Bashkirian and lower Moscovian (Vereian) fusulines (van Ginkel 1965; Wagner 1971, and a number of more recent publications by various researchers) (Fig. 2). This observation is confirmed by recent studies from the Santo Firme and La Camocha Mine sections, where latest Bashkirian and earliest Moscovian fusulines (Villa & Merino-Tomé 2016) were identified from a stratigraphic interval yielding Langsettian micro- and macroflora (Horvarth 1985; Wagner & Álvarez-Vázquez 1995, 2010), and also by the age of tonstein TC-1 (Fig. 6). In addition, the Duckmantian megaflora from the Curavacas Formation in Los Cintos (locality number 59 of Wagner & Álvarez-Vázquez 2010) correlates with the *Profusulinella* B subzone of van Ginkel (1965) (late Vereian–early Kashirian in age). These data could document a potential diachronism between the base of the Duckmantian and Bolsovian substages in the Cantabrian Zone with respect to other regions of the world.

There seem to be fewer difficulties in correlating paralic and/or continental areas in the upper Bolsovian as well as in the Asturian, Cantabrian and Barruelian substages. The three last units were introduced in the Cantabrian Zone on the basis of their megafloral content (Wagner & Winkler Prins 1985*a*,*b*; Wagner *et al.* 2002). Available data suggest that the megafloral zones characterizing these substages are relatively isochronous (Fig. 6). In the Donets Basin, the base of the *Linopteris bunburii* Zone, which is equivalent to *Linopteris obliqua* Zone of the Asturian substage, is located in the m_3 coal seam (Opluštil *et al.* 2016*a*). The stratigraphic unit containing the m_3 coal seam is considered to belong to the middle Podolian (Fissunenko & Laveine 1984; Izart *et al.* 1996; 1998; Davydov *et al.* 2010, 2012), a stratigraphic position that, according to fusuline data, would fall within the Caleras stratal package of the Central Asturian Coalfield, where the base of the Asturian substage is placed (Wagner & Álvarez-Vázquez 1991) (Fig. 2).

On the other hand, the base of the *Odontopteris cantabrica* Zone, which characterizes the Cantabrian substage (Wagner 1984; Wagner & Álvarez-Vázquez 2010), correlates with the uppermost Myachkovian in both the Cantabrian Zone (Sánchez de Posada *et al.* 2002*b*; Wagner & Álvarez-Vázquez 2010) and the Donets Basin (n_1^1 coal seam, Opluštil *et al.* 2016*a*; underlying the N_2 limestone, Izart *et al.* 1998; Davydov *et al.* 2010, 2012). This level seems to be well constrained on the basis of the geochronological dates yielded by tonstein TC-5 (307.1 ± 1 Ma) and n_1 (307.26 ± 0.1 Ma; Davydov *et al.* 2010) and sample 13 (305.99 ± 0.07 Ma; Opluštil *et al.* 2016*a*) in the Bohemian Basin (Czech Republic) (Fig. 6).

There is an apparent close correspondence in the age of the base of the *Alethopteris zeilleri* Zone (Saberian) in the Donets Basin (estimated as 304 Ma by Davydov *et al.* 2010) and Bohemia (303.7 Ma; Opluštil *et al.* 2016*a*). This age is in accordance with the age obtained in the present study for the TC-6 tonstein (304.2 ± 1.1 Ma) (Fig. 6). Moreover, the CA-TIMS ages (302.25 ± 0.28 and 302.13 ± 0.23 Ma) yielded by two tonsteins of Saberian age in the Sabero Coalfield (Cantabrian Zone) (Knight & Wagner 2014), are coincident with the age estimated for the *Alethopteris zeilleri* Zone in the Donets Basin and Bohemia (see Opluštil *et al.* 2016*a*).

Relevance of the new LA-ICP-MS dates in the Cantabrian Zone and potential contribution to global correlation and to the geological timescale

In spite of the problems derived from the provincialism of the Carboniferous faunas, the global correlation of the Pennsylvanian subsystem has been improved thanks to the effort of the Subcommission on Carboniferous Stratigraphy task groups (Villa & Task Group 2004; Groves & Task Group 2007; Heckel *et al.* 2007; Ueno & Task Group 2009; Nemirovska *et al.* 2010; Alekseev & Task Group 2013; among many other contributions). Nevertheless, further research is still needed for selecting palaeontological markers of the stage and substage boundaries and the subsequent GSSPs of the Pennsylvanian units (e.g. Richards 2013). This remains a major challenge, for which three main limitations are evident, as follows.

(1) Most of the stratotypes of the marine Pennsylvanian stages (Serpukhovian, Moscovian, Kasimovian and Gzhelian) and substages were established in successions of the Russian Platform (see Davydov *et al.* 2012), which are known to

exhibit numerous unconformities caused by subaerial exposure during glacioeustatic sea-level falls (Kabanov 2003; Kabanov & Baranova 2003; Kabanov *et al.* 2016). The chronostratigraphic boundaries are often coincident with lithostratigraphic boundaries and these usually correspond to significant unconformities with subaerial erosion surfaces and palaeosol development (Ross & Ross 1987, 1988; Alekseev *et al.* 1996; Kabanov 2003; Kabanov & Baranova 2003). As a consequence, these successions are likely to contain significant gaps in the fossil record.

(2) The provincialism of the marine Carboniferous faunas challenges the global correlation of the marine Pennsylvanian strata. As an example, the correlation of the Moscow Basin succession with two very relevant Carboniferous areas in the Urals and the Donets Basin is not straightforward, and is still under debate. However, the precise correlation of stages and substage boundaries of the Pennsylvanian successions integrated in the stratigraphic composite standards used for GST2012 must be supported on a biostratigraphic framework.

(3) The correlation of the stages and substages of the West European chronostratigraphic scale, in particular those defined in non-marine strata, such as the Asturian, Cantabrian, Barruelian, Saberian and Stephanian B, with the East European stages defined in the Russian Platform presents some difficulties.

To solve these problems, it will be necessary to achieve a reliable integration of biostratigraphic and radiometric data from as many well-documented successions as possible, and from different areas of the world. In this context, the Cantabrian Zone foreland basin, situated near the equator throughout the Carboniferous and characterized by high subsidence rates and sedimentation through the Pennsylvanian in both marine and paralic environments, has great potential to contribute to worldwide correlation. However, essentially owing to the lack of precise radiometric ages, the valuable biostratigraphic information from the Cantabrian Zone has not been included in any of the composite standards used to build the GST2012. Our LA-ICP-MS radiometric dates offer the possibility to include this information in further updated versions of the Geological Time Scale. A composite standard of the Cantabrian Zone could eventually be constructed by using these radiometric ages, or new higher precision dates by means of ID-TIMS. In this regard, it is relevant to mention that the analysed volcanic ash-fall layers yielded a high number of zircons with concordant ages (>50%), indicating that they are good candidates for future ID-TIMS dating.

Conclusions

Robust radiometric ages have been obtained by means of LA-ICP-MS dating of seven ash-fall layers interbedded within the Carboniferous successions of the Cantabrian Zone (northern Spain). Taking into account the analytical uncertainty, the ages obtained show a good match with the inferred ages estimated for each sample on the basis of the available biostratigraphic information on conodonts (samples BA-1 and BA-2), fusulines (samples TC-1, TC-3, TC-4 and TC-5) and megaflora (samples TC-3, TC-4, TC-5 and TC-6). Nevertheless, it is relevant to mention that the date of the weighted-mean ages of samples TC-1, TC-4 and TC-5 appears, in these cases, to be slightly younger (*c.* 0.3 – 1 Ma) than expected from the biostratigraphic framework, but still within the estimated error ranges. The fact that in each of the seven samples there are a very high number of zircons with concordant magmatic (i.e. related to the volcanic event) ages indicates that most of the samples should be suitable for future ID-TIMS dating.

The new LA-ICP-MS radiometric dates also offer the possibility of considering valuable biostratigraphic information from the Cantabrian Zone to construct future geological time scales. Eventually, a composite standard of the Cantabrian Zone could be constructed using these preliminary radiometric ages or new high-precision ages by means of ID-TIMS. The latter will be relevant in the case of sample TC-1 (314.4 ± 1.3 Ma), located within the Bashkirian–Moscovian transition interval in the Cantabrian Zone, along with the uppermost Moscovian (mid to upper Myachkovian) samples TC-3 (307.7 ± 1.3 Ma), TC-4 (307.1 ± 1 Ma) and TC-5 (307.1 ± 1 Ma), which can offer key dates for future estimations of the absolute ages of the Bashkirian–Moscovian and Moscovian–Kasimovian boundaries. Also, in the case of sample TC-1, it could serve for, first, corroborating a potential diachronism of the first appearances of *Verella*, *Eofusulina* and other fusuline species, which could be younger in the Cantabrian Zone than in the Donets Basin, an observation that would support the interpretation of Davydov (2009) that first occurrence of certain fusuline taxa took place earlier in the Donets Basin than in other regions, and, second, to investigate the potential diachronism of Langsettian floras, which could be younger in the Cantabrian Zone than in other areas of the world according to the age of sample TC-1 together with the fact that attributed Langsettian floras were contemporaneous in the Cantabrian Zone with lower Moscovian (Vereian) fusulines.

Acknowledgements S. Opluštil and I. Villa are gratefully acknowledged for their constructive reviews, which substantially improved the paper. J. Prado and A. Gómez Borrego are warmly thanked for their invaluable help and enthusiastic collaboration, which allowed us to obtain the tonstein samples TC-1, TC-2 and TC-6 stored in the Instituto Nacional del Carbón (INCAR-CSIC). Sampling of tonsteins from the Agapita, Lozanita and 2nd Refugio coal seams was possible thanks to the facilities provided by Hunosa Mining Company and the collaboration of M. Cámara, N. Canto Toimil, P. Fuente and A. Alonso. We also wish to thank J. R. Bahmonde and L. P. Fernández, who participated in the study of the Las Baleas Quarry. Assistance by A. Gärtner, U. Linnemann and R. Kraus at the Senkenberg Museum Dresden is greatly appreciated, and we thank A. López-Carmona for collaboration in sample analysis.

Funding The authors acknowledge financial support from the Spanish Ministry of Economy and Competitiveness under projects CGL2013-46061-P, CGL2013-44458-P and CGL2016-76438-P (to J.F.-S.), project SV-PA-13-ECOEMP-45 and FEDER funding from the European Community.

Scientific editing by Igor Villa

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